

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :  
RALF NOERENBERG, ET AL. : EXAMINER : RUSSEL J. KEM-  
MERLE III  
SERIAL NO: 10/559,493 :  
FILED: DECEMBER 05, 2005 : GROUP ART UNIT: 1791  
FOR: METHOD FOR INCEASING THE CUTTING HARDNESS OF A MOLDED  
BODY

DECLARATION

I, Sebastian Ahrens, hereby declare and state that I am a Ph.D., having received my doctorate in Chemistry from the TU Dresden, Germany,

That I am employed by BASF SE in Ludwigshafen, Germany,

That I have been working in several departments of R & D from 04/2004 up to now. Since 11/2007 I am working on catalysts, especially on crystalline aluminosilicates,

That I am the author or co-author of 10 patent applications in different fields of chemistry including catalysts and ethylene amines,

That I have read and believe to have understood the Office Action dated January 15, 2009,

That the following experiments were made in accordance to my specific instructions under my personal supervision and that the results were as stated below:

## Experiment 1

### Experiment 1:

Two H-ZSM-5 catalysts were prepared, having a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio of 1000 to 1. The catalysts were dried overnight at 120 °C and subsequently calcined at temperatures of 500 °C for 5 hours. Catalyst 1 (comparative example) was directly used in a process for preparing a triethylenediamine (TEDA) by reaction of ethylenediamine (EDA) and piperazine (PIP) according to claim 11 as presently on file.

Catalyst 2 (according to the invention) was treated with water vapour at 175 °C for 24 hours. Subsequently catalysts 2 was used in a process according to claim 11.

The results are shown in table 1.

The treatment according to the invention leads to an increase of the EDA and PIP conversion from 97 % (EDA)/49 % (PIP) to 99 % (EDA)/50% (PIP). Moreover the TEDA selectivity increases from 93 % up to 95 %.

Table 1

Catalyst [a]	H <sub>2</sub> O [b]	Modulus [c]	Reaction Conditions [d]		
			Conversion (PIP) [%]	Conversion (EDA) [%]	Selectivity (TEDA) [%]
1	No	1000	97	49	93
2	Yes	1000	99	50	95

[a] H-ZSM-5

[b] 175 °C,  
24 h

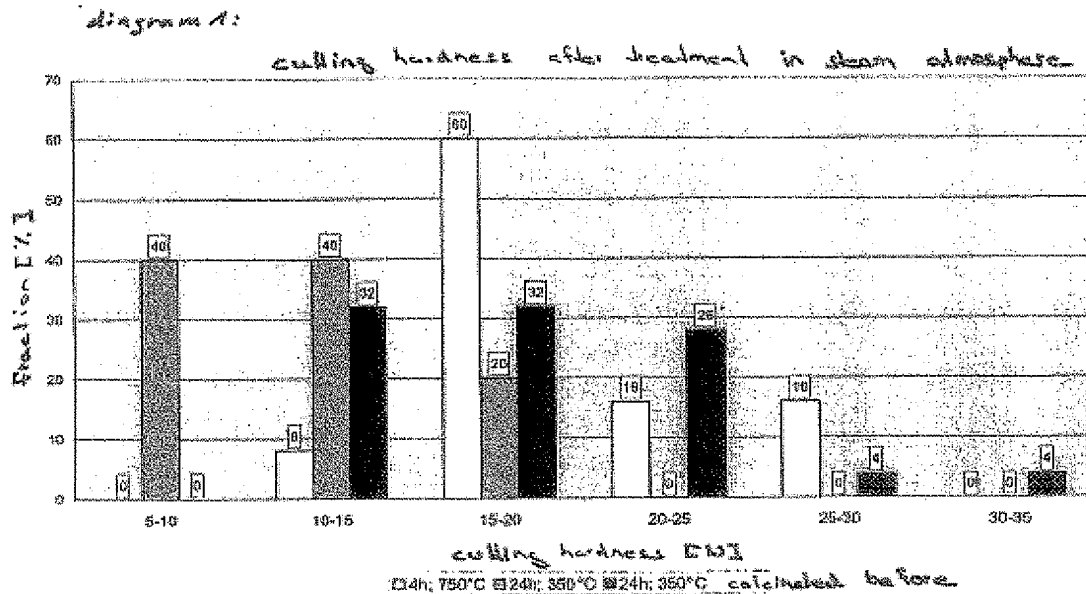
[c]  
SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>

[d] WHSV = 0.5 g/(g h), T = 350 °C, EDA/PIP/H<sub>2</sub>O =  
25/25/50

## Experiment 2

A mixture consisting of 20 wt.-% SiO<sub>2</sub>-binder and 80 wt.-% mordenit as powder (crystalline aluminosilicate, H-form, module: 13) was extruded into shaped bodies of 3 mm size.

Afterwards, the following experiments were carried out: 1 kg of the shaped bodies was filled into a rotary furnace. Afterwards, the shaped bodies were treated with steam (WHSV = 1 kg(H<sub>2</sub>O)/kg(Kat)h). After cooling down to room temperature, the cutting hardness was measured according to the method described in the present application (page 11, lines 1 – 11). For every treatment 25 shaped bodies were investigated. The distribution of the cutting hardness is shown in the following figure.



Gray columns: i) 24 h at 350 °C (in steam atmosphere);

White columns: ii) 4 h at 750 °C (in steam atmosphere) according to Ogawa, column 7, catalyst preparation example 1;

Black columns: iii) Calcinated: 5 h at 500 °C (without steam atmosphere) in a muffle furnace, then 24 h at 350 °C (in steam atmosphere) according to the present invention.

I declare that all statements made in this declaration of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that wilful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of

Title 18 of the United States Code and that such wilful false statements may jeopardize the validity of the application or any patent issuing thereon.

Ludwigshafen, April 01, 2003.

Sebastian Ahrens  
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